



THE  
ONTARIO WATER RESOURCES  
COMMISSION  
  
ANALYTICAL SERVICES  
  
of the  
  
OWRC LABORATORY DIVISION

MOE  
ANA  
ANPF

c.1  
a aa

### Copyright Provisions and Restrictions on Copying:

This Ontario Ministry of the Environment work is protected by Crown copyright (unless otherwise indicated), which is held by the Queen's Printer for Ontario. It may be reproduced for non-commercial purposes if credit is given and Crown copyright is acknowledged.

It may not be reproduced, in all or in part, for any commercial purpose except under a licence from the Queen's Printer for Ontario.

For information on reproducing Government of Ontario works, please contact ServiceOntario Publications at [copyright@ontario.ca](mailto:copyright@ontario.ca)

ANALYTICAL SERVICES OF THE OWRC LABORATORY DIVISION

	<u>Page</u>
INTRODUCTION .....	1
DESCRIPTION OF BRANCHES - Bacteriology .....	1
Biology .....	2
Chemistry I .....	2
Chemistry II .....	3
SUBMISSION OF SAMPLES - Shipping .....	3
Bacteriology .....	4
Biology .....	4
Chemistry .....	5
PERISHABILITY - Bacteriology .....	6
Biology .....	6
Chemistry .....	7
INTERPRETATION OF RESULTS - Bacteriology .....	8
Biology .....	8
Chemistry .....	8
PRECISION AND ACCURACY .....	8
COSTS .....	9

## INTRODUCTION

The Ontario Water Resources Commission conducts a water quality management program to provide water of the highest quality throughout Ontario and to reduce water pollution to the lowest possible level. In addition to legal, financial, scientific and engineering aspects, the program requires analytical capability to:

- a) assess the quality of provincial water supplies;
- b) define the type and level of pollution associated with waste discharges; and
- c) provide data to support requirements for remedial action where OWRC objectives are not met.

The responsibility for providing these analytical services lies with the four branches of the Division of Laboratories: Bacteriology, Biology, Chemistry I and Chemistry II. The Division is equipped to do all standard water quality tests, as well as some special analyses required for research studies and unique pollution problems. These services may be used by staff members and other public agencies engaged in water quality management.

The main laboratories are located in Toronto, with field laboratories now in operation at London and the Lakehead. Mobile laboratories are used in conjunction with major surveys in areas remote from the regular laboratories.

## DESCRIPTION OF BRANCHES

### Bacteriology Branch

The Bacteriology Branch is primarily concerned with the analysis of water from municipal distribution systems, surface and ground water supplies, and waste water disposal systems, to ensure the maintenance of satisfactory water quality.

Coliform analysis is a routine test for drinking water samples, and the recently developed presence-absence (P-A) test, which detects the presence of coliform, fecal coliform, fecal streptococcus, pseudomonas and clostridium bacteria, has now been introduced as a routine test.

Samples collected for investigation of taste, odour and colour problems are analyzed for iron, sulphur and other pertinent bacteria. "Plate counts" and other bacterial analyses are performed to meet the requirements of particular problems or surveys.

The Branch operates two mobile laboratories which can be set up close to the site of a major survey.

### Biology Branch

The programs of the Biology Branch are designed to evaluate the presence or absence of pollution, using biological parameters, and to determine the effects of pollution on aquatic life. These programs include biological surveys, investigations of algae problems at water treatment plants, fish bioassay and toxicity tests, evaluation of aquatic pesticides and the issuance of permits for aquatic nuisance control measures.

The Branch conducts complete surveys, including collecting the samples. However, most tests are carried out routinely and samples may be submitted by Commission field staff. The routine analytical techniques include identification and enumeration of algae, identification of bottom fauna and vascular plants, threshold odour determinations, fish bioassays, taste tests (fish tainting) and pesticide analyses.

The Branch regularly identifies specimens of fish, aquatic insects, worms, etc., collected by field staff in the course of their work.

### Chemistry I Branch - Water and Pollution Analysis

The basic function of this Branch is the chemical analysis of water samples from domestic supplies, surface waters and domestic waste, but excluding those associated with industrial waste discharges. The Branch is equipped to do large numbers of each of the standard water quality tests.

Samples from municipal water supplies are examined for the various chemicals making up their mineral content, including hardness, alkalinity, chloride, fluoride, iron and other inorganic components.

The characteristics most often examined in cases of polluted water and waste discharges are nitrogen compounds; phosphorus; conductivity, turbidity and solids content; and oxygen demand tests, either chemical or biochemical.

The procedures given in "Standard Methods for the Examination of Water & Wastewater" are usually followed but are often adapted to facilitate the use of automated analytical equipment. New methods and tests are periodically developed which have general application to the analytical needs of the Commission, but the Branch does not have the resources to develop specific procedures of limited applicability.

Sewage sludges and natural water sediments are normally accepted for analysis for common chemical constituents.

## Chemistry II Branch - Industrial Waste Analysis

This Branch provides chemical analyses of waste water, surface water, sediments and aquatic biota, to detect and identify industrial waste materials.

Industrial wastes contain a great variety of compounds, and the Branch has routine methods available for over one hundred different analyses, including nearly all of the metals, many inorganic compounds and a wide variety of organic substances. This great variety of tests is achieved at the expense of the number of determinations which can be made, since many of the methods are complex and time consuming and do not lend themselves to automation.

Several pesticides can be measured in water and waste waters and the Branch is performing the analyses required in monitoring the DDT levels in fish.

The Branch conducts chemical analyses of the organic compounds extracted from carbon filters placed on the Great Lakes and some of their tributaries.

The unique aspects of industrial waste problems often demand considerable analytical methods development work and this constitutes a major part of the Branch's activities.

### SUBMISSION OF SAMPLES

Careful attention must be given to the details of sampling and sample handling prior to arrival at the laboratories in order to ensure that a representative sample in good condition is provided for examination. A number of booklets are provided by the Commission which give detailed instructions for sampling for specific purposes. Some examples are:

Acceptance of Samples for Analyses

Drinking Water Objectives

Investigation of Fish Kills

Shipping Procedures

This guide draws attention to the general details of sampling procedures which directly affect the laboratory staff in successfully completing the analyses.

### Shipping

Sample containers for all tests are obtained from the Stores Branch in the laboratory building in Toronto and from field

laboratories. A list of the available types of bottles may be obtained on request.

Each container is accompanied by the appropriate submission forms which must be completed in legible writing. Shipments must be clearly marked as to the number of samples and the number of boxes, etc., and then sent by the fastest route. Exposure to extreme temperatures can cause deterioration of the samples and must be avoided.

Requests for containers should be limited to the minimum actually needed. Occasionally, container shortages develop due to many of them being idle rather than being in transit. Unused containers should be returned as quickly as possible.

If a survey ends unexpectedly, please inform the shipping staff immediately so that no more empty containers are sent. Failure to do this results in costly delays in having them returned by the shipping companies.

#### Bacteriology

Sterile six-ounce (180 ml) bottles must be used to collect samples for submission to the OWRC laboratory for bacterial analysis. Samples from distribution systems should be taken from taps only after water has been flowing for two minutes. The samples should be collected directly into sterile bottles, not by means of a dipper or other container. An air space should be left in the bottle unless other instructions are given.

Unusual sampling conditions must be noted on the bottle following prior consultation with the laboratory staff. Bottles containing sodium thiosulphate are available and should be used when taking samples from chlorinated supplies.

#### Biology

The collection of aquatic biota for analysis often requires special tools such as nets and dredges, and, for this reason, the Branch generally conducts its own sample collection. It is recommended that those wishing to submit samples should contact the laboratory to clarify the procedures to be used for the particular investigation and to determine whether the laboratory has the capacity to do the test at that time. Many biological analyses are very time consuming, and advance warning of samples is very helpful in planning laboratory operations.

It is very important to include with the samples a complete description of the problem and the type of information required.

Forty-ounce bottles normally hold enough sample for any one of the following: phytoplankton count, threshold odour determinations and fish bioassay tests. This may only be enough to determine the relative toxicity; the amount required for a more detailed evaluation can vary from as little as a cupful up to 40 gallons.

Special procedures are required for collecting fish for pesticide analysis and taste tests, and programs involving these tests should be coordinated with the laboratory staff before sampling begins.

Details of sampling methods for investigating fish kills are given in the booklet mentioned above.

### Chemistry

The method of collecting the sample in the field is the concern of the individual in charge of the particular survey; however, care must be taken to prevent contamination by sampling devices, motor exhaust, disturbing the bottom, etc. Water samples are normally placed in 32 or 40-ounce glass bottles, although plastic bottles must be used for tests which are adversely affected by contact with glass. The Division has information available on the use of special containers and it is in the best interest of the sampler to enquire before collecting the sample. This is particularly important in industrial waste analyses.

When the sample is sent to the laboratory, a detailed description of known constituents should be included on the submission sheet, particularly if there is an unusual one, such as an industrial waste in a water sample. Most of the tests are subject to interferences (reactions which produce false results) produced by substances other than the one being measured. Very often, these effects can be eliminated or reduced by taking the proper precautions; however, it requires that the sampler give prior indication of possible contaminants. For example, a BOD determination is often meaningless if the sample contains material toxic to bacteria. If no warning is given, then the test will be carried out normally and an incorrect result will be reported.

If a group of related samples is submitted, a map of the area, or some indication of the location of waste inputs, is very helpful to the laboratory staff. It is also a good practice to number the samples in some logical order, such as downstream in a river. All analyses are examined before being released and an anomalous result might be improperly rejected as being incorrect, whereas some details of the location would confirm its validity.

The exact analyses required must be stated. Samples cannot be accepted with such requests as "chemical analysis" or "all the metals". If there is doubt as to what analyses to request,



a brief description of the purpose of collecting the sample will enable the laboratory staff to apply some appropriate tests.

When requesting analyses of sediments and for organic compounds, it is important to define the purpose of the test. In most cases, there is more than one analytical technique available and each may measure a slightly different component. The choice will depend on the purpose of the test.

Samples which separate into more than one phase are particularly troublesome unless the request is very clear as to whether the second phase is to be included in the analysis or not. For example, if the request is for total phosphorus and there is a layer of sludge in the bottle, the analyst must decide whether to try and mix the solids in or not, and, in fact, might not do what the sampler wants. In cases of multiphase samples, the required tests should read "including solids" or "water phase only", etc.

Samples containing volatile material must be tightly sealed and unmistakably marked.

For unusual analyses, particularly those of industrial origin, an indication should be given as to whether quantitative or qualitative results are required. For the protection of the analyst, samples containing cyanide or other toxic materials should be clearly marked.

#### PERISHABILITY

##### Bacteriology

Samples left for some time before being analyzed undergo changes (increases or decreases) in bacterial population, depending upon the initial bacterial density, the presence of growth-promoting or toxic substances in the water and the environmental conditions under which the samples are held during shipment to the laboratory. Analyses should be performed the same day that samples are collected, but if chilled, usually no drastic changes in population occur within a 24-hour period.

Samples received after 48 hours may be analyzed, but, because of changes in the bacterial population, interpretation of results is usually not done. Samples received after four days are rejected. Negative coliform results for drinking water samples arriving more than 48 hours after collection may be incorrect and fresh samples should be collected.

##### Biology

Biological samples are often very perishable and special precautions apply to each type.

Phytoplankton samples must be preserved with Lugols Iodine solution (enough to impart a dark orange colour to the sample) which is available from the laboratory.

Vascular aquatic plants must be placed in plastic bags and kept cool and moist.

Samples of fish for taste tests and chemical analysis should be quick frozen and the boxes clearly marked "Frozen Fish - Keep Cool". Samples of fish, insects, algae, etc., collected for identification only should be preserved in 5% formaldehyde or 80% ethanol if they cannot be submitted in a fresh condition.

Water samples for threshold odour determinations must arrive at the laboratory within 36 hours of collection and no preservatives can be added.

### Chemistry

The concentrations of almost all components undergo some changes with time after collection. This may be due to biological activity in the sample, chemical precipitation, adsorption or desorption with the walls of the container, or even chemical reaction between constituents.

A number of common substances are so perishable that analysis must be carried out in the field at the time of collection. Examples of such tests are dissolved oxygen, hydrogen sulphide, carbon dioxide and chlorine. Analytical data for several other components such as ammonia, soluble phosphorus and soluble iron must be regarded with some doubt when there is a delay of more than a few hours between sampling and analysis.

Three general methods for reducing the perishability are: proper selection of sample container, reduced temperature during transit, and rapid delivery to the laboratory.

Preservation methods are constantly being examined, and the laboratory staff is often able to advise samplers on what methods are best suited for a particular purpose. In cases where no information is available regarding a component of critical importance, some preliminary investigations should be conducted, but this must be done well in advance of starting the sampling program.

A major difficulty in preserving samples is that while some parameters are stabilized, others may be adversely affected. For example, freezing samples immediately after collection will preserve nitrogen and phosphorus parameters but it alters the conductivity of the sample.

## INTERPRETATION OF RESULTS

Staff members from all branches are available for discussion regarding the analytical methods used and the meaning of any results. The Laboratory may be contacted either in writing or by telephone at 416-248-3421.

### Bacteriology

Results for potable water supplies are returned along with an indication of suitability for drinking purposes.

Analyses carried out as part of an investigation of a specific problem or of a field survey may or may not be interpreted, depending on the requests of the sampler.

### Biology

Analytical results are normally returned as a report which includes as complete an interpretation as possible.

### Chemistry

While the results of routine tests are normally reported without any comment as to their meaning, evaluation and interpretation will be given if requested. The Chemistry II Branch regularly prepares detailed reports on method applied and the meaning of results pertaining to special industrial waste problems.

## PRECISION AND ACCURACY

The precision of an analytical method is a measure of the difference between multiple determinations made on a single sample.

The accuracy of a method is a measure of the difference between the observed result and the correct value.

An analytical procedure may be very precise but not accurate, and an imprecise method may give accurate results by doing enough replications.

The precision and accuracy of all tests are constantly being examined and improved. The precision of most tests is known and is obtained by doing multiple analyses on individual samples. The accuracy of many tests is not known. When it is possible, instruments and methods are calibrated using solutions of known concentrations. However, it is known that for samples collected in the field there may be interferences and other factors which cause a precise and calibrated method to give inaccurate results and there is no way of eliminating this phenomenon.

When available, precision data is given in "Standard Methods" or on the analytical outlines available from the laboratory division. This data only applies to laboratory work; variability in sampling and perishability are not included. Both of these factors may introduce a much greater error than any arising in the laboratory.

#### COSTS

The analytical services are supported by the regular OWRC budget and are available to other government agencies without charge.

The average laboratory operating costs for each test are of the order of one dollar for the high volume test carried out by Chemistry I Branch, three dollars for bacteriological tests, eight dollars for the industrial waste determinations, and fourteen dollars for the biological analyses. The average cost for many specific tests is several times the over-all average, and for any analysis the cost can increase a great deal if special techniques are required. In cases where analytical methods have to be developed, the cost per test may be several hundred dollars or more.

In the interest of economy, samplers should only request those tests which are relevant to the particular problem. When requesting non-routine analyses, the importance of the results should be weighed against the potential very high cost.

9183

En

pf

MOE/ANA/ANPF  
Ontario Ministry of the En  
Analytical services  
of the OWRC laboratory anpf  
division c.1 a aa